Selective dimerization of higher cycloolefins in the presence of micro- and micromesoporous zeolite catalysts*

N. G. Grigor 'eva, * S. V. Bubennov, A. N. Khazipova, L. M. Khalilov, and B. I. Kutepov

Institute of Petrochemistry and Catalysis, Russian Academy of Sciences, 141 prosp. Oktyabrya, 450075 Ufa, Russian Federation. Fax: +7 (347) 231 2750. E-mail: ngg-ink@mail.ru

Selective synthesis of dimers of cycloolefins C_6-C_8 was carried out in the presence of highly dispersed zeolite catalysts HY, HBeta, and HZSM-12 and granulated zeolite HY-WB, which differ in acidic properties and pore structure. The high selectivity of microporous zeolite HZSM-12 in cyclohexene dimerization (100%) and micromesoporous zeolite HY-WB in cycloheptene and cyclooctene dimerization (90–95%) was established.

Key words: cycloolefins, cyclohexene, cycloheptene, cyclooctene, dimerization, microand micromesoporous zeolites.

Cyclic olefins are traditional and accessible raw materials for organic and petrochemical synthesis. Cyclene oligomers find diverse practical use as solvents, plasticizers,¹ and components of drugs and cosmetic materials.^{2–4}

Oligomerization of the most studied compound, cyclohexene, in the presence of homogeneous acidic catalysts P_2O_5 , BF_3 , H_2SO_4 , HF, and AlCl₃ (see Refs 5–11) affords a mixture of di-, tri-, tetra-, and higher-molecularweight oligomers. The dimeric fraction, obtained by cyclohexene oligomerization in the presence of H_2SO_4 , contains 2- and 3-methylcyclopentylcyclohexanes along with 1-(1'-cyclohexenyl)cyclohexane (**1a**).^{8,11} This indicates that sulfuric acid induces the isomerization of one of the rings.

In the presence of the metathesis catalysts, cyclohexene can transform itself into cyclic unsaturated compounds, *viz.*, dimers and trimers (catalyst WCl₆), or into saturated and aromatic hydrocarbons (catalytic system WCl₆ + ROH + EtAlCl₂, where R = Et, Ph, PhCh₂).¹²

It was established^{13,14} by the study of cyclohexene transformations in the presence of the heterogeneous catalysts that chromium, zirconium, and vanadium oxides, manganese protoxide, and other catalysts exhibit different activities in cyclohexene dehydrogenation to benzene. Such catalysts as SiO₂, Al₂O₃, BeO; mixed silica, alumina, thorium, or zirconia; or aluminosilicates favor the isomerization of the six-membred ring into the five-membered ring.^{13–17} In addition to cyclohexene isomerization, polymerization and hydrogen redistribution occur. In this process, hydrogen donors are intermediate compounds leading to coking and hydrocarbons of the polymer fraction.

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Cyclohexene is transformed into cyclohexane, 1-, 2-, and 3-methylcyclopentenes, methylcyclopentane, and dimers on granulated zeolite Y modified by rare-earth metal (REM) cations.¹⁸ The yield of dimers is independent of the concentration of the REM cation, whereas the yield of the hydrogen transfer products (cyclohexane and methylcyclopentane) increases with an increase in the REM content from 2.1 to 13.0%. The hydrogen transfer reaction is favored by the use of dealuminated Y zeolites with the molar ratio SiO₂/Al₂O₃ (*M*) > 30.¹⁹

The Mobil Oil Corporation patented the method for the dimerization of cycloolefins C_5-C_{12} on zeolites ZSM-12 (M > 83) in an autoclave at 75–400 °C.²⁰ It was shown that the olefin conversion decreases and the selectivity of dimer formation increases with an increase in the ring size.

The synthesis of dimers of other higher cyclenes is poorly studied. The preparations of the cyclooctene dimer, *viz.*, 1-(1'-cyclooctenyl)cyclooctane (2a), in the presence of sulfuric acid²¹ and of a mixture of two cyclooctene dimers 2a,b in the presence of nickel acetylacetonate²² (Scheme 1) were described.



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Catalyst	Degree	М*	Equilibriu		Acidic properties				
	of crystallinity (arb.%)		capacity $P/P_{\rm s} = 0.8$	capacity at 20 °C and $P/P_s = 0.8$ for vapors of		_x /°C	Concentration of acid sites /µmol g ⁻¹		
			water	benzene	Ι	Π	CI	C_{II}	C _{I+II}
HY	100	6.0	0.30	0.30	250	400	622	560	1182
HBeta	100	18.0	0.26	0.32	280	450	530	340	870
HZSM-12	100	34.0	0.15	0.16	300	450	409	260	669
HY-WB	85	7.6	0.23	0.27	250	420	515	410	925

Table 1. Physicochemical characteristics of the zeolite catalysts

* Molar ratio SiO₂/Al₂O₃.

The purpose of the present work is to develop selective catalytic methods for synthesis of dimers of cyclohexene, cycloheptene, and cyclooctene in the presence of zeolite catalysts with micro- (HY, HBeta, HZSM-12) micro-mesoporous (HY-WB) structures.

Results and Discussion

Characterization of zeolite catalysts. Zeolite catalysts of various structural types with different acidic properties and porous structures were used. Zeolites HY, HBeta, and HZSM-12 have a microporous structure, whereas in zeolite HY-WB (without binders) the microporous structure of individual crystals combines with meso- and macropores formed between aggregates of zeolite crystals.^{23,24} According to the data of mercury porosimetry, the specific surface of meso- and macropores of this zeolite is $8.5 \text{ m}^2 \text{ g}^{-1}$, and their volume is $0.32 \text{ cm}^3 \text{ g}^{-1}$. Thus, meso- and macropores structure of the granules thus providing efficient diffusion of molecules of reacting substances to the catalytically active sites.

The physicochemical properties of the studied zeolites are given in Table 1. According to the X-ray diffraction data and adsorption characteristics of the zeolites, all samples of microporous zeolites are characterized by the degree of crystallinity close to 100%. For the sample of zeolite Y-WB, the relative crystallinity is \sim 85%.

The spectra of zeolite acidity obtained by temperatureprogrammed desorption (TPD) of ammonia contain two peaks characterizing weak acid sites with the temperature maximum (T_{max}) at 100–350 °C and strong acid sites with T_{max} in the range >350 °C. The total concentration of acid sites maximizes in zeolite HY and decreases on going to zeolite HY-WB and further to high-silica zeolites HBeta and HZSM-12. The strength of acid sites as estimated by the shift of the temperature maximum of the peak in the TPD spectrum, decreases in the following order: zeolite HBeta = HZSM-12 > HY-WB > HY.

Dimerization of cyclic olefins. The composition of the oligomerization products of cycloolefins depends on the chemical structure of the monomer, properties of the zeo-lite catalysts, and reaction conditions.

In the presence of the zeolite catalysts, cyclohexene is mainly transformed into dimers (87.9-100%), among which 1-(1'-cyclohexenyl)cyclohexane (1a) prevails (Scheme 2). In addition to compound 1a, other cyclohexene dimers are also observed: unsaturated 1-(2'-cyclohexenyl)- (1b) and 1-(3'-cyclohexenyl)cyclohexane (1c) and saturated bicyclohexane (1d).

At temperatures higher than 150 °C, oligomerization is complicated by destructive processes leading to methylcyclopentenes, methylpentenes, and hexadienes. In the



Scheme 2



presence of the studied zeolites, these compounds, as the starting olefins, undergo oligomerization to form a complex mixture of products.

Along with cyclohexene dimers, higher-molecularweight oligomers, trimers and tetramers, can be formed on the zeolite catalysts. According to the data of gas chromatography coupled with mass spectrometry (GC/MS), trimers include, together with compounds with the molecular weight (MW) 246, hydrocarbons with MW 242 and 244, formation of which is possible due to the hydride ion transfer reactions (Scheme 3). The occurence of hydrogen redistribution reaction as indicated by the presence of saturated hydrocarbons (1d) and aromatic compounds in the reaction mixture is characteristic of olefin conversion on the acid zeolites.²⁵

A comparison of the catalytic properties of zeolites of various structural types (Table 2) shows that the most active catalyst is zeolite HBeta, in the presence of which the conversion of cyclohexene attains 65-74% (150-200 °C). The higher activity in the reaction of zeolites HBeta and HY compared to that of zeolite HZSM-12 is due, most likely, to the high concentration of acid sites in the former

two catalysts and lower steric hindrances for transport of the monomers and dimers in the zeolite pores. Actually, the pore size in zeolite HY is 0.75 nm, in zeolite HBeta it is 0.64-0.75 nm, and the pores of zeolite HZSM-12 are smaller (0.55-0.67 nm).²⁶

Dimer **1a** is most selectively (up to 100%) formed on the catalyst HZSM-12 at 100–120 °C (10–20 wt.% of the catalyst). The selectivity of formation of dimer **1a** is lower (76–82 and 94–96%, respectively) on zeolites HY and HBeta under the same conditions, because the dimerization products also contain compounds **1b**–**d**. The temperature increase to 180–200 °C leads to a more complex composition of the dimeric fraction, since it contains, along with dimers **1a–d**, hydrocarbons obtained by the di- and codimerization of products of cyclohexene destruction. The broadest fraction of dimers was obtained on zeolite HY, in the presence of which isomerization and destruction of cyclohexene and hydrogen transfer occur most intensely. A possible explanation is the heterogeneity of the acid sites of HY zeolite.²⁷

The total yield of the dimers decreases on zeolites HY and HBeta with the temperature increase to 200 °C, but

Catalyst	C _{cat} (wt.%)	<i>Т</i> /°С	X (%)		Oligomers (%)						
					Dimers				Tetra-		
				1a	1b,c	1d	Other		mers		
HY	20	120	20.7	75.7	10.0	6.5	4.7	3.1	_		
	20	180	50.0	55.1	19.9	9.1	6.7	9.1	0.1		
	20	200	56.3	52.4	18.6	9.6	9.8	9.4	0.2		
HBeta	10	120	61.4	95.8	2.9	0.1	0.8	0.4	_		
	20	100	47.0	93.8	3.0	_	1.8	1.4	_		
	20	120	61.7	89.5	3.0	0.5	4.4	2.6	_		
	20	150	64.9	80.1	13.3	1.4	2.9	2.3	_		
	20	200	74.3	58.7	16.4	1.3	11.0	12.1	0.5		
HZSM-12	10	120	5.0	100.0	_	_	_	_	_		
	20*	100*	40.7	100.0	_	_	_	_	_		
	20	150	14.5	92.4	6.7	0.9	_	_	_		
	20	180	33.2	85.3	8.9	2.9	2.9	—	—		

Table 2. Oligomerization of cyclohexene in the presence of the zeolyte catalysts (nonane, initial monomer concentration in the solvent $[M_0] = 1.2 \text{ mol } L^{-1}$, 5 h)

Note. Here and in Tables 3 and 4, C_{cat} is the amount of the catalyst and X is conversion.

* The experiment was carried out for 16 h.

the amount of higher-molecular-weight oligomers, trimers and tetramers, increases (see Table 2). Only cyclohexene dimers are formed by cyclohexene oligomerization in the presence of zeolite HZSM-12.

The results of transformations of cycloheptene and cyclooctene in the presence of the zeolite catalysts are given in Tables 3 and 4.

A comparison of the conversion of cycloolefins on diverse zeolites shows that zeolite HBeta exhibits the highest activity.

Cycloheptene forms dimers on all microporous zeolites, but their composition changes substantially depending on the reaction conditions. At 40–60 °C more than 90% of the dimer composition fall on the fraction of two compounds, **3a,b**, formed in a ratio of ~1 : 1 (Scheme 4).

At 120–180 °C cycloheptene undergoes structural and destructive transformations to form cyclic and alicyclic compounds, namely, methylcyclohexenes and methyl-cyclohexanes, dimethylcyclopentenes, isoheptenes, *etc.* As a result, the number of compounds in the dimeric fraction increases sharply.

In the ¹³C NMR spectrum of dimer **3b**, the quaternary carbon atoms at the double bond arranged between two cycloheptane rings are characterized by a signal in a low field at δ 133.28. Due to high symmetry of the molecule,



the C atoms of the methylene groups in both cycloheptane moieties appear as three intense signals at δ 31.42, 28.88, and 27.97, which exhibit (in HSQC experiment) direct C—H bonds with the corresponding protons of the CH₂ groups at δ 2.25, 1.57, and 1.52 (see Experimental).

Cyclooctene isomerization on zeolites HY, HBeta, and HZSM-12 starts already at 40 °C and, hence, the reaction products contain a large amount of hydrocarbons with the empirical formula C_8H_{14} and dimers, among which compounds **2a**,**b** predominate (their structure is shown in Scheme 1).

Catalyst	C _{cat} (wt.%)	<i>T</i> /°C	Solvent	X (%)	Composition (%)			
					Isomers	Dimers 3a,b	Other products	
НҮ	20	60	_	4.2	3.1	88.1	8.8	
	20	120	Undecane	23.6	16.8	39.4	43.8	
HBeta	20	100	Undecane	48.3	19.5	48.5	32.0	
	20	150	Undecane	62.1	12.4	40.2	47.4	
HZSM-12	20	100	_	8.9	1.8	54.5	43.7	
	20	120	_	15.4	10.1	36.1	53.8	
HY-WB	10	40	_	55.2	9.8	90.2	_	

Table 3. Transformations of cycloheptene in the presence of the zeolyte catalysts ($[M_0] = 1.2 \text{ mol } L^{-1}$, 5 h)

Table 4. Transformations of cyclooctene in the presence of the zeolyte catalysts ($[M_0] = 1.2 \text{ mol } L^{-1}$, 5 h)

Catalyst	C _{cat} (wt.%)	<i>T</i> /°C	Solvent	X (%)	Composition (%)			
					Isomers	Dimers 2a,b	Other products	
HY	10	60	Undecane	6.2	58.1	25.1	16.8	
	10	120	Undecane	34.1	48.8	20.5	30.7	
	20	120	Undecane	40.8	41.3	20.6	35.1	
HBeta	20	40	_	15.2	38.2	35.9	25.9	
	50	40	_	33.2	24.1	44.1	31.8	
	100	40	_	40.3	10.4	40.8	48.8	
	10	120	_	83.2	35.7	22.9	41.4	
HZSM-12	20	60	_	5.1	71.8	14.7	13.5	
	10	120	_	68.2	53.4	16.1	30.5	
	30	180	_	95.9	56.7	13.2	30.1	
HY-WB	10	60	_	50.4	5.9	94.1	—	

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The content of products 2a,b in the dimers does not exceed ~60% even at 40—50 °C. As the temperature increases, the yield of hydrocarbons obtained by the dimerization and codimerization of compounds, which are products of the destructive transformations of cyclooctene, increases.

The yield of dimers maximizes on zeolite HBeta and decreases on zeolites HY and HZSM-12.

It was shown for cyclooctene transformation in the presence of zeolite HBeta that the conversion of the monomer and the yield of dimers increased with an increase in catalyst concentration. This dependence is characteristic of reactions that occur on the external surface of catalysts.

The data obtained indicate that the possibilities of dimerization of cycloheptene and cyclooctene in the presence of zeolites with microporous structure are constrained by the molecular-sieve properties of the catalysts. The application of zeolite HY-WB with the combined micromesoporous structure in the dimerization of the ole-fins made it possible to synthesize dimers of cycloheptene (**3a**,**b**) and cyclooctene (**2a**,**b**) with high selectivity 90-94% (see Tables 3 and 4). The amount of isomerization products does not exceed 10%.

Thus, the study of the catalytic properties of the zeolite catalysts with different acidic properties and characteristics of the porous structure made it possible to develop selective methods for synthesis of dimers of cycloolefins C_6-C_8 . It was found that zeolites HY and HBeta with the maximum concentration of strong acid sites exhibit the highest activity in cyclohexene dimerization. However, a high acidity of these catalysts favors the intense occurrence of isomerization and destruction reactions, which finally results in a more complex composition of the reaction products. The major product of cyclohexene isomerization is 1-(1'-cyclohexenyl)cyclohexane, whose selectivity of formation on zeolite HZSM-12 achieves 100%.

The dimerization of cycloolefins C_7-C_8 with retention of the ring structure on the microporous zeolite catalysts is impeded because steric hindrances appear when the bulky molecules of monomers and dimers move inside the catalyst channels. The dimers of cycloheptene (**3a**,**b**) and cyclooctene (**2a**,**b**) were synthesized with a selectivity of 90-94% in the presence of zeolite HY-WB with the micromesoporous structure.

Experimental

Cycloolefins (cyclohexene, cycloheptene, cyclooctene) purchased from Acros were used as received. Prior to experiments the solvents (nonane, undecane) were purified using standard procedures.²⁸

Highly dispersed zeolites Y, Beta, and ZSM-12 were studied, as well as granulated zeolite Y without binders (Y-WB) in the H form. Zeolites HY with the degree of exchange of Na⁺ ions by H⁺ (α) equal to 96% and HY-WB (α = 94%) were obtained by the decationation of zeolites NaY (M = 6) and NaY-WB (M = 5), which were synthesized using known procedures,^{23,29} respectively. Zeolites NH_4 -Beta (M = 18) and NH_4 -ZSM-12 (M = 34), produced at the OAO "Angarskii zavod katalizatorov i organicheskogo sinteza" (Angarsk Plant of Catalysts and Organic Synthesis, Angarsk, Russia), were transformed into the H form by calcination at 540 °C. Prior to catalytic tests, the zeolite samples were subjected to the thermal treatment in air for 4 h at 540 °C.

The physicochemical characteristics of the zeolite catalysts were determined using described procedures.^{24,30}

The conversion of the initial monomers and the composition of the reaction products were determined by GLC and HPLC as described previously.³¹

Dimers of compounds 1–3 were identified using GL/MS and 1D and 2D NMR spectroscopy. High-resolution mass spectra were recorded on a Fisons instrument with chromatograph equipped with a DB-560 quartz column (50 m); the column was heated from 50 to 320 °C with a rate of 4 °C min⁻¹; an electron impact of 70 eV was applied. ¹H and ¹³C NMR spectra were recorded on a Bruker AVANCE-400 spectrometer with working frequencies of 400.13 and 100.62 MHz, respectively, in standard tubes (5 mm in diameter) for solutions of substances in CDCl₃. 2D Homo- (COSY HH) and heteronuclear (HSQC, HMBS) correlation experiments were performed using pulse field gradient procedures.

1-(1'-Cyclohexenyl)cyclohexane (1a). Cyclohexene (5 mL, 0.048 mol) and zeolite HZSM-12 (0.8 g) were loaded in a glass tube. Then the tube was sealed and placed inside a metallic autoclave, which was continuously rotated in a temperaturemaintained bond for 5 h at 150 °C. After cooling and separating the catalyst, 3.9 g of the product was obtained. The product had the following composition (wt.%): cyclohexene, 59.3; isomers, 0.8; 1-(1'-cyclohexenyl)cyclohexane, 38; other dimers, 1.7; trimers, 0.2. 1-(1'-Cyclohexenyl)cyclohexane (1a) was isolated from the obtained mixture under reduced pressure. The yield was 38%, b.p. 60 °C (40 Torr). Found (%): C, 87.25; H, 12.75. C₁₂H₂₀. Calculated (%): C, 87.73; H, 12.27. ¹H NMR, δ: 1.13 (m, 1 H, C(4)H₂); 1.18 (m, 2 H, C(2)H₂, C(6)H₂); 1.29 (m, 2 H, C(3)H₂, C(5)H₂); 1.55, 1.59 (both m, 1 H each, C(4')H₂); 1.60 (m, 1 H, C(4)H₂); 1.64 (m, 1 H, C(5')H₂); 1.68 (m, 2 H, C(2)H₂, C(6)H₂); 1.72 (m, 1 H, C(1)H); 1.75 (m, 1 H, C(5')H₂); 1.77 (m, 2 H, C(3)H₂, C(5)H₂); 1.78 (m, 1 H, C(3')H₂); 1.94 (m, 1 H, C(6')H₂); 2.00 (m, 1 H, C(3')H₂); 5.39 (s, 1 H, C(2')H). ¹³C NMR, δ: 22.85 (C(5')); 23.23 (C(4)); 25.27 (C(3')); 26.50 (C(4)); 26.75 (C(6'));26.85 (C(3), C(5)); 31.92 (C(2), C(6)); 45.88 (C(1)); 119.34 (C(2')); 142.34 (C(1')). MS, m/z: 164. Kovats index: 1329.

1,1'-**Bicyclohexane (1d).** The counter synthesis of bicyclohexane was carried out by the hydrogenation of 1-(1'-cyclohexenyl)cyclohexane (1a) on Pd/Al₂O₃ in an autoclave for 6 h at 150 °C. After cooling and separating the catalysts, product 1d was isolated from the reaction mixture under reduced pressure, b.p. 79.7 °C (5 Torr). Found (%): C, 85.91; H, 14.09. C₁₂H₂₂. Calculated (%): C, 85.67; H, 14.33. ¹H NMR, δ : 1.02 (m, 4 H, C(2)H₂, C(2')H₂, C(6)H₂, C(6')H₂); 1.08 (m, 2 H, C(1)H₂, C(1')H₂); 1.25 (m, 6 H, C(3')H₂, C(4')H₂, C(4')H₂, C(5')H₂); 1.70 (m, 2 H, C(4)H₂, C(4')H₂); 1.71 (m, 4 H, C(2)H₂, C(5')H₂), C(6')H₂), 1.72 (m, 4 H, C(3)H₂, C(3')H₂, C(5)H₂), C(5')H₂). ¹³C NMR, δ : 26.93 (C(3), C(3'), C(4), C(4'), C(5), C(5')); 30.21 (C(2), C(2'), C(6), C(6')); 43.52 (C(1), C(1')). MS, *m/z*: 166. Kovats index: 1319.

1-(1⁻-Cycloheptenyl)cycloheptane (3a) and 1,1⁻-bicycloheptylidene (3b). Dimers 3a,b were synthesized similarly to compound 1a in the presence of zeolite HY-WB for 10 h at 110 °C. The composition of the obtained oligomerizate is the following (wt.%): cycloheptene, 30; isomers, 10; dimers, 60. Dimers 3a,b were isolated under reduced pressure. The total yield was 58.3%, b.p. 145-150 °C (4 Torr). ¹H NMR of compound 3a (subtracted from the spectrum of the mixture), δ : 1.40–1.60 (m, 14 H, C(3)H₂, C(4)H₂, C(5)H₂, C(6)H₂, C(3')H₂, C(4')H₂, C(5')H₂, C(6')H₂); 1.74 (m, 2 H, C(2')H₂, C(7')H₂); 2.02 (m, 1 H, C(1')H); 2.06 (m, 2 H, C(7)H₂); 2.09 (m, 2 H, C(3)H₂); 5.54 (t, 1 H, C(2)H, J = 6.4 Hz). ¹³C NMR, δ : 27.37 (C(3), C(6)); 27.66 (C(6')); 27.85 (C(5')); 28.08 (C(4), C(5)); 28.14 (C(4')); 28.26 (C(7')); 30.80 (C(3')); 33.49 (C(2), C(7)); 50.20 (C(1)); 123.33 (C(2')); 151.28 (C(1')). MS, m/z: 192. Kovats index: 1568.¹H NMR of dimer **3b**, δ : 1.52 (m, 8 H, C(4)H₂, C(5)H₂, C(4')H₂, C(5')H₂); 1.57 (m, 8 H, C(3)H₂, C(6)H₂, C(3')H₂, C(6')H₂); 2.25 (m, 8 H, C(2)H₂, C(7)H₂, C(2')H₂, C(7')H₂). ¹³C NMR, δ: 27.97 (C(4), C(5), C(4'), C(5')); 28.88 (C(3), C(6), C(3'), C(6'); 31.42 (C(2), C(7), C(2'), C(7')); 133.28(C(1), C(1')). MS, *m/z*: 192.34. Kovats index: 1589.

1-(1'-Cyclooctenyl)cyclooctane (2a) and 1,1'-bicyclooctylidene (2b). Dimers 2a,b were synthesized similarly to compounds **3a,b**. The composition of the oligomerizate is as follows (wt.%): cyclooctene, 17; isomers, 31.7; dimers, 51.3. Dimer 2b was isolated by crystallization from ethanol from a mixture of dimers 2a,b, which was obtained by vacuum distillation. The crystals were washed with cold ethanol and dries. The yield of mixture 2a,b was 48.8%, b.p. 140-150 °C (5 Torr). ¹H NMR of compound 2a, δ: 0.95–1.71 (m, 26 H, CH₂); 2.00 (m, 1 H, C(9)H); 5.34 (m, 1 H, C(2)H, J = 16.0 Hz). ¹³C NMR, δ : 26.24 (C(4), C(6)); 26.64 (C(3), C(7)); 27.04 (C(8')); 28.55 (C(5'), C(6')); 29.80 (C(4'), C(7')); 29.85 (C(3')); 32.07 (C(2), C(8)); 46.50 (C(1)); 121.47 (C(2')); 147.32 (C(1')). MS, m/z: 220.39. Kovats index: 1814. Dimer 2b, cr.p. 30 °C. Found (%): C, 86.35; H, 13.65. C₁₆H₃₀. Calculated (%): C, 86.89; H, 13.11. ¹H NMR, δ: 1.48 (m, 12 H, C(4)H₂-C(6)H₂, C(4')H₂-C(6')H₂); 1.64 (m, 8 H, C(3)H₂, C(7)H₂, C(3')H₂, C(7')H₂); 2.21 (m, 8 H, $C(2)H_2$, $C(8)H_2$, $C(2')H_2$, $C(8')H_2$). ¹³C NMR, δ : 26.24 (C(4)-C(6), C(4')-C(6')); 27.25 (C(3), C(7), C(3'), C(7'));31.16 (C(2), C(8), C(2'), C(8')); 132.97 (C(1), C(1')). MS, *m/z*: 220.39. Kovats index: 1823.

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